

# **Laboratory Analysis**



Wisconsin Department of Agriculture, Trade and Consumer Protection Division of Food and Recreational Safety

datcp.wi.gov

## Introduction

Wisconsin State Statute, s. 97.34(2)(e), requires the Department of Agriculture, Trade and Consumer Protection to compile and publish an annual bottled drinking water report. It is a compilation of the laboratory results for bottled drinking water sampled in the State of Wisconsin in Fiscal Year 2016. The samples were collected from bottled water processors and retail distribution sites in this state. The collection and analysis is a coordinated effort by the Bureau of Food Safety and Inspection, the Bureau of Laboratory Services and the Wisconsin Laboratory of Hygiene.

There are 18 bottled drinking water processors in the State of Wisconsin. They are licensed under the category "Food Processing Plant" by the Department of Agriculture Trade and Consumer Protection and are required to comply with the same quality and safety standards as municipal water systems. They are licensed and regulated under Wisconsin Administrative Codes:

- Chapter ATCP 70 "Food Processing Plants"
- Chapter NR 809 "Safe Drinking Water"
- Section NR 140.10 "Groundwater Quality"

To provide reasonable assurance of compliance with state health-related standards, bottling establishments have specific requirements for product sampling, analysis, record keeping, and reporting. They test for bacteria each month, nitrates every quarter, volatile organics, pesticides, and inorganics every third year, and radionuclides every five years. The processor maintains the results of microbiological analysis for a year, chemical analysis for six years, and radiological analysis for ten years.

In the state fiscal year ending June 30, 2016, Eighteen 2-gallon samples of commercial bottled water from seventeen licensed establishments were collected and analyzed. These samples included water from both 'private' wells and municipal water sources. The samples were analyzed for substances that cause either aesthetic defects or are contaminants of public health concern. The substances and their regulatory limits are included in the Enforcement Standards tables starting on page 5.

All 2016 samples except for one met current public health enforcement standards (s. NR 140.10, Wis. Adm. Code). One sample was positive for coliform bacteria. Coliform bacteria, when detected, is/are an indicator that the water supply is 'at risk' for contamination. When the coliform was detected, a follow-up investigation was conducted and re-sampling occurred to assure that water to be bottled was free of coliform.

Two samples did not meet Secondary, or Aesthetic Standards. Those samples contained dissolved solids levels that were above the enforcement limit, but as the standard is an aesthetic standard only, no follow-up action was needed for a sample with the total dissolved solids as reported.

#### Background

There is no such thing as naturally pure water. In nature, all water contains some impurities. As water flows in streams, accumulates in lakes, and filters through layers of soil and rock in the ground, it dissolves or absorbs many of the substances that it touches. Some of these substances are harmless. In fact, some people prefer mineral water precisely because minerals give it an appealing taste. However, at certain levels, just like man-made chemicals, minerals may be considered contaminants that can make water unpalatable or even unsafe.

#### Man-made Contaminants

Man-made contaminants may also affect water that is bottled. These contaminants may be substances discharged from factories, applied to farmlands, or used by consumers in their homes and yards. Microbiological and chemical contaminants can enter water supplies. These materials can be the result of human activity or can be found in nature. For instance, chemicals can migrate from disposal sites and contaminate sources of drinking water. Coliform bacteria from human and animal wastes may be found in drinking water if the water is not properly treated or disinfected.

These bacteria are used as indicators that other harmful organisms may be in the water. If coliform bacteria are found in a water sample, further testing is conducted to see if there are any fecal or pathogenic bacteria present.

Water naturally contains less than 1 milligram of nitrate-nitrogen per liter. When higher levels are present, it indicates that the water has been contaminated. Common sources of nitrate contamination include fertilizers, animal wastes, septic tanks, municipal sewage treatment systems, and decaying plant debris. State and federal laws set the maximum allowable level of nitrate-nitrogen in public drinking water at 10 milligrams per liter.

#### **Natural Contaminants**

Naturally occurring contaminants can also be found in drinking water. Some contaminants come from erosion of natural rock formations. Groundwater, that moves slowly through the pores or cracks in underground layers of rock, dissolves minerals as it travels. Dissolved solids are minerals or salts that have been dissolved in the water while in the aquifer. Dissolved solids can be calcium, magnesium, salt, iron or other minerals. Some of these dissolved solids may actually come out of solution during storage if the water becomes cold enough or if some of the water evaporates. These precipitates are not harmful, but may not be appealing to the consuming public.

Fluoride can be added to water supplies to promote healthy teeth. It can also be present in water from the erosion of natural deposits or discharge from fertilizer and aluminum factories.

Water can also pick up naturally occurring radium or man-made radionuclide as it flows to the water source. The radioactive gas radon-222 occurs in certain types of rock and can leach into ground water. Most rock contains some radium, usually in small amounts. Testing for radionuclide is a relatively recent change in DATCP requirements. The testing process for water samples begins with a screening for "gross alpha/beta particle activity" which measures the total amount of one type of radioactivity given off by the water. If high levels of gross alpha/beta activity are found, further testing for radium is conducted. Radioactivity levels are measured in "pico curies" per liter of water (abbreviated "pCi/L").

In Wisconsin, most of the community water supplies which exceed the radium standard draw water from a deep sandstone aquifer and are located in a narrow band which stretches from Green Bay to the Illinois state line. In addition, a few high radium levels have been found in groundwater from sandstone formations in west central Wisconsin and in granite formations in north central Wisconsin. In all cases, the radium was present in the rock and water long before the first well was drilled.

## **Bottled Water Regulation**

On May 13, 1996 new bottled water regulations from FDA took effect. The new regulations were aimed at alleviating consumer confusion about the many different types of bottled water on the market by providing standard definitions for the terms "artesian water," "ground water," "mineral water," "purified water," "sparkling bottled water," "spring water," "sterile water," "well water," and others.

Bottled water, like all other foods regulated by FDA, must be processed, packaged, shipped and stored in a safe and sanitary manner and be truthfully and accurately labeled. Bottled water products must also meet specific FDA quality standards for contaminants. Since 1996, mineral water must also meet the bottled water standards. Mineral water had previously been exempt from standards that applied to other bottled water. FDA web site: www.fda.gov.

## FDA has established the following definitions:

Bottled Water: Water that is intended for human consumption and that is sealed in bottles or other containers with no added ingredients except that it may contain safe and suitable antimicrobial agents.

Artesian Water or Artesian Well Water: Water from a well tapping a confined aquifer in which the water level stands at some height above the top of the aquifer.

Ground Water: Water from a subsurface saturated zone that is under a pressure equal to or greater than atmospheric pressure.

Mineral Water: Water containing not less than 250 parts per million total dissolved solids, originating from an underground water source. No minerals may be added to this water.

Purified Water: Water that is produced by distillation, deionization, reverse osmosis or other suitable processes and that meets the definition of "purified water" in the U.S. Pharmacopeia, 23d Revision, January1, 1995.

Sparkling Bottled Water: Water that, after treatment and possible replacement of carbon dioxide, contains the same amount of carbon dioxide that it had at emergence from the source.

Spring Water: Water derived from an underground formation from which water flows naturally to the surface of the earth.

Well Water: Water from a hole bored, drilled, or otherwise constructed in the ground, which taps the water of an aquifer.

In addition to defining these terms, the regulation addresses various other labeling concerns. For example, water bottled from municipal water supplies must be clearly labeled as "from a community water system" or, alternatively, "from a municipal source", unless it is processed sufficiently to be labeled as "distilled" or "purified" water.

The regulation also requires accurate labeling of bottled water marketed for infants. If a product is labeled "sterile" it must be processed to meet FDA's requirements for commercial sterility. Otherwise, the labeling must indicate that it is not sterile and should be used in preparation of infant formula only as directed by a physician or according to infant formula preparation instructions.

## Summary of Results FY 2016

## **Enforcement Standards for Safety**

- Of eighteen samples tested one was positive for coliform bacteria.
- Of eighteen samples tested six tested positive for trace amounts of one or more pesticides.
- Of eighteen samples tested six samples contained n-nitrate/nitrites at levels above sensitivity but within the acceptable standards. The enforcement level for n-nitrate/nitrites is 10 mg/l.
- Eighteen samples were tested for radionuclide activity. All of the results were well below the regulatory limit. Seven of the results were 'negative,' meaning they had less activity than background activity.

Contaminants	Number Positive	Percent Positive	Average Value of Positive Results	Enforcement Level	Percent of Regulatory Limit
N-nitrate/nitrite	6	33	3.08 mg/L	10 mg/L	31
Coliform Bacteria	1	6	>1/100 mL	1/100 mL	100
Pesticides	6	33	0.05 μg/L	3 μg/L	2
Radionuclides					
Gross Alpha	15	83	0.9 pCi/l	15.0 pCi/L	6
Gross Beta	14	78	1 pCi/L	50 pCi/L	2

## **Quality and Aesthetic Standards**

Analyses were conducted for secondary inorganic and physical standards (s. NR 809.60, Wis. Adm. Code). These address the aesthetic quality of drinking water such as taste, odor and appearance.

- Sixteen of Eighteen samples were below the enforcement standards. Two contained total dissolved solids that exceeded enforcement standards.
- Twelve samples contained detectable levels of chloride, two samples contained detectable levels of fluoride, no samples contained detectable levels of copper, one sample contained detectable levels of zinc, and fifteen samples contained detectable levels of sulfates. All detectable levels were well below enforcement standards.
- Dissolved solids were detected in fifteen of nineteen samples. No samples tested above the
  enforcement level for total dissolved solids standards. There are no adverse health effects from
  dissolved solids providing that the sulfate level is also low. Since the level of sulfates was below
  enforcement levels in this sample, there is no cause for concern. At the most, the dissolved solids
  could cause an 'off' flavor.

Contaminants	Number Positive	Percent Positive	Average Value of Positive Results	Enforcement Level	Percent of Regulatory Limit
Chloride	12	67	21.9 mg/L	250.0 mg/L	9
Copper	ND	NA	NA	1.0 mg/L	NA
Fluoride	2	11	0.386 mg/L	2.0 mg/L	19
Sulfates	4	22	0.050 mg/L	250.0 mg/L	20
Zinc	1	6	0.016 mg/L	5.0 mg/L	0.32
**Dissolved Solids	15	83	216 mg/L	500.0 mg/L	43

<sup>\*\*</sup>For the 2 samples that exceeded the enforcement level for total dissolved solids, the percent of regulatory limit was 118%

## Public Health Groundwater Quality Standards and Test Sensitivities<sup>1</sup>

Items noted with an \* were not detected in any of the samples

Test	Enforcement Standard	Test Sensitivity
Coliform	No Presence	1/100 mL
Simazine	4.0 μg/L	0.050 μg/L
Carbofuran*	40.0 μg/L	0.051 μg/L
Lead*	15.0 μg/L	0.50 μg/L
Cyanides*	200 μg/L	10 μg/L
Endrin*	2.0 μg/L	0.50 μg/L
Lindane*	0.2 μg/L	0.50 μg/L
Toxaphene	3.0 μg/L	1.0 μg/L
Methoxychlor	40.0 μg/L	0.50 μg/L
2,4-D*	70.0 μg/L	0.050 μg/L
2,4,5-TP*	50.0 μg/L	0.050μg/L
N-Nitrate/Nitrite	10.0 mg/L	0.50 mg/L
Atrazine	3.0 μg/L	0.050μg/L
De-ethyl atrazine	3.0 μg/L	0.050 μg/L
De-isopropyl atrazine*	3.0 μg/L	0.050 μg/L
Di-amino atrazine	3.0 μg/L	0.28 μg/L
Gross Alpha	15.0 pCi/L	Sample Dependent
Gross Beta	50.0 pCi/L	Sample Dependent

## Secondary Chemical & Physical Standards<sup>2</sup>

Test	Enforcement Standard	Test Sensitivity
Dissolved Solids	500.0 mg/L	10 mg/L
Chlorides	250.0 mg/L	2.0 mg/L
Copper	1.0 mg/L	20 μg/L
Fluoride	2.0 mg/L	0.20 mg/L
Iron	0.3 mg/L	50 μg/L
Sulfates	250.0 mg/L	10 μg/L
Zinc	5.0 mg/L	10 μg/L
Arsenic <sup>3</sup>	10 μg/L	1.5 μg/L

mg/L = Milligrams per Liter ug/L = Micrograms per Liter MPN/100mL = Most Probable Number in 100 milliliters pCi/L = picocuries/liter of water

<sup>&</sup>lt;sup>1</sup> From NR 140 Register, December 2010 & From NR 809 Register, March 2016

<sup>&</sup>lt;sup>2</sup> Aesthetic Standard from NR 809.60 Register, March 2016

<sup>&</sup>lt;sup>3</sup> Maximum Contaminant Level (MCL) for Arsenic enforceable after April 4, 2016

#### Some Informational Web sites:

## **EPA**

- http://www.epa.gov/safewater/dwhealth.html "Drinking Water and Health"
- http://www.epa.gov/safewater/dwh/contams.html
   "Information About Water Contaminants"

## Wisconsin

- The Wisconsin DNR Drinking Water and Ground Water web site
- Wisconsin Department of Agriculture, Trade & Consumer Protection rule web site Chapter 97 Food Regulation

Producer	Units of Measure	Croix Crystal Water Treatment	Premium Waters Inc.	Wisconsin Glacier Springs	Century Springs Bottling Company	Mineral Spring Water LLC
License Number		309813-F2	287769-F2	121770-F2	212344-F2	254714-F2
Location		Hudson	Chippewa Falls	New Berlin	Mukwonago	Oshkosh
Sample Number		1	2	3	4	5
Amount Sampled		2 gallons	2 gallons	2 gallons	2 gallons	2 gallons
Sample Date		Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016
Test						
Endrin	μg/L	<0.50	<0.50	<0.050	<0.50	<0.50
Fluoride	mg/L	<0.20	<0.20	<0.20	<0.20	0.22
Lindane	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Methoxyclor	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Nitrogen-Nitrate/Nitrite	mg/L	<0.50	4.08	<0.50	0.645	<0.50
Atrazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
2,4,5-TP	μg/L	< 0.050	<0.050	<0.050	<0.050	<0.050
Simazine	μg/L	< 0.050	<0.050	<0.050	<0.050	<0.050
Sulfates	μg/L	<10	<10	<10	<10	91.8
Toxaphene	μg/L	<1.0	<1.0	<1.0	<1.0	<1.0
Carbofuran	μg/L	<0.051	<0.051	<0.051	<0.051	<0.051
Chlorides	mg/L	<2.0	23.7	26.2	30.7	96
Cyanide	μg/L	<10	<10	<10	<10	<10
De-ethyl Atrazine	μg/L	<0.050	<0.050	<0.050	0.003-0.050	<0.050
Deisopropyl Atrazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Diamino Atrazine	μg/L	<0.28	<0.28	<0.28	<0.28	<0.28
2,4-D	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Total Dissolved Solids	mg/L	<10	160	92	164	620
Arsenic	μg/L	<1.5	<1.5	<1.5	<1.5	<1.5
Copper	μg/L	<20.0	<20.0	<20.0	<20.0	<20.0
Iron	μg/L	<50.0	<50.0	<50.0	<50.0	<50.0
Lead	μg/L	<0.5	<0.5	<0.5	<0.5	<0.5
Zinc	μg/L	<10	<10	<10	<10	<10
Detect Coliform with Colisure	/100 mL	<1	<1	<1	<1	<1
E. coli	/100 mL	No test needed	No test needed	No test needed	No test needed	No test needed
Gross alpha	pCi/L	0.213	1.58	0.319	0.009	3
Gross beta	pCi/L	-0.226	2.01	0.944	1.28	2.08

Producer	Units of Measure	Wisconsin Springs Country Valley	Valley Springs Artesian Gold LLC	Hayden Water Company dba Markey Springs	Twigs Beverage	Culligan Water Conditioning
License Number		121522-F2	122209-F2	207848-F2	121490-F2	296687-F2
Location		Ontario	Portage	Luxemburg	Shawano	Burlington
Sample Number		6	7	8	9	10
Gallons Sampled		2 gallons	2 gallons	2 gallons	2 gallons	2 gallons
Sample Date		Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal year 2016
Test						
Endrin	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Fluoride	mg/L/L	<0.20	<0.20	<0.20	<0.20	<0.20
Lindane	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Methoxyclor	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Nitrogen-	mg/L	<0.50	2.87	2.13	3.86	<0.50
Atrazine	μg/L	<0.050	0.008-0.050	<0.050	<0.050	<.050
2,4,5-TP	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Simazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Sulfates	μg/L	<10	<10	<10	18.9	<10
Toxaphene	μg/L	<1	<1	<1	<1	<1
Carbofuran	μg/L	<0.051	<0.051	<0.051	<0.051	<0.051
Chlorides	mg/L	<2	11.5	3.75	25.7	7.5
Cyanide	μg/L	<10	<10	<10	<10	<10
De-ethyl Atrazine	μg/L	<0.050	0.136	<0.050	0.003-0.050	<0.050
Deisopropyl Atrazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Diamino Atrazine	μg/L	<0.28	0.088-0.28	<0.28	<0.28	<0.28
2,4-D	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Total Dissolved	mg/L	292	276	104	364	20
Arsenic	μg/L	<1.5	<1.5	<1.5	<1.5	<1.5
Copper	μg/L	<20.0	<20.0	<20.0	<20.0	<20.0
Iron	μg/L	<50.0	<50.0	<50.0	<50.0	<50.0
Lead	μg/L	<0.5	<0.5	<0.5	<0.5	<0.5
Zinc	μg/L	<10	<10	<10	<10	<10
Detect Coliform with	/100 mL	>1.0	<1.0	<1.0	<1.0	<1.0
E. coli	/100 mL	<1	No test needed	No test needed	No test needed	No test needed
Gross alpha	pCi/l	0.067	1.2	0.295	1.07	-0.298
Gross beta	pCi/l	2	0.225	0.558	1.52	-0.162

Producer	Units of Measure	Roundy's Supermarkets Inc	Artesian Wells	Hillestad Pharmaceuticals	Ledgerock Springs	Kwik Trip Dairy
License Number			305367-F2	122142-F2	121895-F2	55-65
Location		Kenosha	Plymouth	Woodruff	Greenleaf	La Crosse
Sample Number		11	12	13	14	15
Gallons Sampled		2 gallons	2 gallons	2 gallons	2 gallons	2 gallons
Sample Date		Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016
Test						
Endrin	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
luoride	mg/L	<0.20	<0.20	<0.20	<0.20	<0.20
_indane	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Methoxyclor	μg/L	<0.50	<0.50	<0.50	<0.50	<0.50
Nitrogen-Nitrate/Nitrite	mg/L	<0.50	<0.50	<0.50	<0.50	<050
Atrazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
2,4,5-TP	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Simazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Sulfates	μg/L	<10	11.1	<10	79.9	<10
Toxaphene	μg/L	<1	<1	<1	<1	<1
Carbofuran	μg/L	<0.051	<0.051	<0.051	<0.051	<0.051
Chlorides	mg/L	<2	3.5	<2	28.2	3
Cyanide	μg/L	<10	<10	<10	<10	<10
De-ethyl Atrazine	μg/L	<0.050	<0.050	0.05	0.003-0.050	<0.050
Deisopropyl Atrazine	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Diamino Atrazine	μg/L	<0.28	<0.28	<0.28	0.088-0.28	<0.28
2,4-D	μg/L	<0.050	<0.050	<0.050	<0.050	<0.050
Total Dissolved Solids	mg/L	24	276	<10	560	20
Arsenic	μg/L	<1.5	1.61	<1.5	<1.5	<1.5
Copper	μg/L	<20.0	<20.0	<20.0	<20.0	<20.0
ron	μg/L	<50.0	<50.0	<50.0	<50.0	<50.0
_ead	μg/L	<0.5	<0.5	<0.5	<0.5	<0.5
Zinc	μg/L	<10	<10	<10	<10	<10
Detect Coliform with	/100 mL	<1	<1	<1	<1	<1
E. coli	/100 mL	No test needed	No test needed	No test needed	No test needed	No test needed
Gross alpha	pCi/l	-0.234	0.139	0.192	3.08	-0.338
Gross beta	pCi/l	-0.002	0.341	0.765	1.86	-1.34

Producer	Units of Measure	Kwik Trip Beverage Plant #369	Genesee Valley Bottling Company	Premium Waters Inc
License Number		226631-F2	121775-F2	121884-F2
Location		LaCrosse	Waukesha	Chippewa Falls
Sample Number		16	17	18
Gallons Sampled		2 gallons	2 gallons	2 gallons
Sample Date		Fiscal Year 2016	Fiscal Year 2016	Fiscal Year 2016
Test				
Endrin	μg/L	<0.50	<0.50	<0.50
Fluoride	mg/L/L	<0.20	<0.20	<0.20
Lindane	μg/L	<0.50	<0.50	<0.50
Methoxyclor	μg/L	<0.50	<0.50	<0.50
Nitrogen-Nitrate/Nitrite	mg/L	<0.50	<0.50	<0.50
Atrazine	μg/L	<0.050	<0.050	<0.050
2,4,5-TP	μg/L	<0.050	<0.050	<0.050
Simazine	μg/L	<0.050	<0.050	<0.050
Sulfates	μg/L	<10	<10	<10
Toxaphene	μg/L	<1.0	<1.0	<1.0
Carbofuran	μg/L	<0.051	<0.051	<0.051
Chlorides	mg/L	3.25	<2.0	<2.0
Cyanide	μg/L	<10	<10	<10
De-ethyl Atrazine	μg/L	<0.050	<0.050	<0.050
Deisopropyl Atrazine	μg/L	<0.050	<0.050	<0.050
Diamino Atrazine	μg/L	<0.28	<0.28	<0.28
2,4-D	μg/L	<0.050	<0.050	<0.050
Total Dissolved Solids	mg/L	12	260	<10
Arsenic	μg/L	<1.50	<1.50	<1.50
Copper	μg/L	<20.0	<20.0	<20.0
Iron	μg/L	<50.0	<50.0	<50.0
Lead	μg/L	<0.500	<0.500	<0.500
Zinc	μg/L	<10	15.8	<10.0
Detect Coliform with Colisure	/100 mL	<1	<1	<1
E. coli	/100 mL	No test needed	No test needed	No test needed
Gross alpha	pCi/l	0.47	6.07	0.128











